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PREDICTION
OF THE
EFFECTS OF STORAGE ON ARP PROPELLANT
BY
MEANS OF CHEMICAL ANALYSIS

BY
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TABLE OF CONTENTS

ABSTRACT

A study of the effects of storage at 65.5° and 80°C on the stabilizer content and viscosity of ARP propellant has been made. The viscosity results were somewhat erratic but, like the stabilizer results, were found to show a definite deterioration trend. Least square analyses of both the viscosity and stabilizer data led to the derivation of simple, linear equations relating these properties with time at either of the temperatures studied. The relationship was extended to any temperature, by application of the Arrhenius equation, so that it is now possible to predict the viscosity or stabilizer content of this type of propellant any desired time at any given storage temperature. The need is shown for obtaining more information concerning the validity of the predictions as well as relating these laboratory tests with storage safety and functioning.

INTRODUCTION

Surveillance of propellants has been conducted at this Arsenal for many years. The objectives of this program are to determine the effects of storage on the safe life and the functioning. Although many stability tests are described in various reports and specifications, their relationship with these objectives has not been clearly demonstrated.

During the past few years, attempts have been made to develop tests that show a regular variation with time. Concurrently, the accumulated stability data is being examined in order to discover the tests that already show such results.

Most recently (Refs. 1,2) it has been shown that the stabilizer content of a propellant could be predicted for any given time, if the storage temperature and the type of propellant were known. These predictions were made possible by the development of new analytical methods (Refs. 3,4) for the propellant stabilizers diphenylamine (DPA) and ethyl centralite (EC).

Since these new methods were more specific than previous methods, analyses made over a period of time were more meaningful. In the past, the analytical methods were simply a measure of unsaturation in the stabilizer, but, with time, this measure became more and more unrelated to the original stabilizer content. As a result of the improved technique, it became possible to follow stabilizer degradation accurately and easily. Studies of propellants stabilized with either DPA or EC indicated that there was a regular pattern of degradation and this pattern was characteristic of the propellant type and storage temperature. From plots of the stabilizer content vs. time, an equation was derived which enabled prediction of stabilizer content at any given time provided that the original concentration and the rate of degradation were known.

Upon being informed of this development, the Quality Assurance Division requested that a similar study be made of the ARP Propellant which is incorporated in the Improved Honest John Rocket, XM50 System. In addition to stabilizer analysis, viscosity measurements were to be made since there was evidence (5-7) that this was also a good measure of degradation. A storage and testing program was set up so that each of the three samples were started at two temperatures (65.5°C and 80°C). At regular intervals portions of these samples were withdrawn and tested for stabilizer content and viscosity. This testing program was continued until the samples fumed, in the case of the higher temperature of storage, and until the supply of samples were used up in the case of the lower temperature of storage.

Samples representing three lots of ARP base grain propellant, designated P-3, P-4, and P-5 respectively, were submitted for the testing program. All were stabilized with 2-nitrodiphenylamine (2NDPA) and had a matrix of nitrocellulose containing 12.6% nitrogen. An analytical method specific for this stabilizer was adapted from one previously developed for DPA.

RECOMMENDATIONS:

1. This type of study should be extended to other propellants. If stabilizers other than DPA, EC or 2NDPA are used, a suitable analytical method should be developed.

2. Further work should be done with this propellant type at other temperatures in order to estimate the reliability of the predictions obtained from the equation.

3. This type of study should be extended to include other tests such as ballistic, calorimetric and closed bomb in order to correlate analytical results with physical tests.

4. Static firing and flight tests should be made in order to determine the relationship between laboratory and functioning tests.

5. A study of viscosity methods should be made in order to permit better measurement of this important property.

CONCLUSIONS:

1. The prediction equation relating stabilizer content with time and temperature has been derived. This equation can be applied to any storage temperature.

2. The test used for determination of viscosity produces results that are erratic but permit derivation of a relationship with time.

3. Further investigations are required to (a) validate these equations (b) establish the relationship with functioning and storability.

RESULTS AND DISCUSSION:

The results obtained on the viscosity measurements are listed in Table I. As shown graphically in figure 1, this data is rather erratic but appears to follow a trend that can best be represented by a straight line. In each case, the line was fitted by the method of least squares and the following equations were obtained:

Lot No.	80°C	65°C
P-3	$Y = -0.016X + 1.45$	$= -0.004X + 1.53$
P-4	$= -0.016X + 1.49$	$= -0.004X + 1.53$
P-5	$= -0.016X + 1.47$	$= -0.001X + 1.46$

In these equations, Y represents relative viscosity while X represents weeks of storage time. As in all linear equations of the form $Y = mx + b$, m is the slope of the line while b is the Y-intercept. In this case the slope indicates the rate of decrease in viscosity while the Y-intercept indicates the viscosity at the start of the test. At 80°C all the samples have the same slope, and the calculated initial viscosities are within five percent of the actual values. At 65°C, the same slope is found for lots P-3 and P-4, but P-5 appears significantly different. The agreement between actual and calculated initial viscosities is within one percent for the first two lots and within 5 percent for P-5.

On the basis of this data, it appears that these equations can be represented in the following generalized form.

$$V_t = kt + V_o \qquad (1)$$

where:

V_t = Relative viscosity at time t.
V_o = Relative viscosity at start of testing program.
k = Reaction rate constant (slope), relative viscosity/wk.
  = -0.016 at 80°C.
  = -0.004 @ 65°C.
t = Storage time, weeks.

Although the results obtained do appear to indicate a trend, the changes are slight so that it is difficult to discriminate between variability due to the testing method and variability due to the effect of storage. For further studies it would be advisable to attempt to modify the method in such a manner as to facilitate the separation of these variables. This could probably be done in several ways. For example, solutions more concentrated than in the present method (7) could be used and the kinematic (6) rather than the relative viscosity could be determined. Investigations of such approaches should lead to a technique that would reveal more clearly the effects of time and temperature.

The results shown in Table II, on stabilizer content, indicated a deterioration trend related to time and temperature that was better defined than the viscosity results. When the data was plotted as shown in figure 2, this regularity was very apparent. As before, a line was fitted to each set of data by the least squares method and the following equations were obtained:

Lot No.	80°C	65.5°C
P-3	$Y = -0.068X + 1.67$	$= -0.014X + 1.62$
P-4	$= -0.072X + 1.82$	$= -0.014X + 1.68$
P-5	$= -0.072X + 1.77$	$= -0.013X + 1.73$

In these equations, Y represents stabilizer content while X represents storage time. For this set of linear equations, the coefficient of X represents the slope of the line while the last number represents the Y-intercept. All slopes are seen to be essentially constant for a given temperature while all the Y-intercepts correspond closely to the stabilizer content at the start of the testing program.

In view of the fact that the slope is a constant for a given temperature and the Y-intercept represents the initial stabilizer content, these equations can be expressed in a generalized form similar to that derived for viscosity.

$$C_t = kt + C_o \qquad (2)$$

where:

C_t = Stabilizer content at time t,%.
C_o = Stabilizer content at start of testing program,%.
k = Reaction rate constant(slope),%/wk.
= -0.072%/wk @ 80°C.
= -0.014%/wk @ 65.5°C.
t = Storage time,weeks.

From equations 1 and 2 it is thus possible to predict the viscosity and stabilizer content of this type of propellant at any time provided that it has been stored at either 65.5°C or 80°C.

Although such equations are helpful, their practical value is limited since the reaction rate constant, k, is known only for the two test temperatures. Obviously, their value would be greatly enhanced if they could be extended to other storage temperatures. This can be done if it is assumed that the decomposition rate varies with temperature while the mechanism remains constant. On the basis of this assumption, the Arrhenius equation (8) can be applied to calculate the energy of activation. This value is a constant which not only characterizes the reaction but also determines the influence of temperature on reaction rate. If the specific rates for two temperatures are known, they can be substituted in the following form of the Arrhenius equation:

$$\log \frac{k_2}{k_1} = \frac{E}{2.3R} \frac{(T_2 - T_1)}{(T_2 T_1)} \qquad (3)$$

where:

T = Absolute temperature, °K.
k = Specific reaction rate constants.
R = Gas constant, energy/degree (Approximately 2 Cal/degree).
E = Activation energy, kilocalories.

This equation can be readily solved for the activation energy, E, if the specific reaction rate constants are the slopes of the lines obtained at 65°C and 80°C for viscosity and stabilizer. When the slopes are substituted in equation 3, the following values are obtained:

Viscosity

$$\log \frac{-0.016}{-0.004} = \frac{E}{2.3(2)} \frac{353 - 338}{(353)\ (338)} \qquad E = 22 \text{ kcal}$$

Stabilizer

$$\log \frac{-0.0072}{-0.0014} = \frac{E}{2.3(2)} \frac{353 - 338}{(353)\ (338)} \qquad E = 26 \text{ kcal}$$

The values obtained for E are in excellent agreement considering the experimental errors and the rounding off done in the calculations. Equation 3 can now be solved for k_2 at any specified temperature. The value thus obtained can be substituted into equations 2 or 3 which can then be solved for V_t or C_t, the viscosity or stabilizer content, after a specific time. In this manner, an estimate of the effects of storage on either of these properties can be made as long as the storage temperature is specified. In other words, either viscosity or stabilizer content, or both, can be predicted for any storage time at any temperature.

Another application of these equations would be to predict the time required for one of these properties to fall to a specified level. Thus, for example, the time required for the stabilizer content of a lot of ARP propellant to fall from an initial value of 1.75% to a quarter of this value when stored at 80°C would be calculated by solving equation 3 for t and substituting:

$$t = \frac{C_t - C_o}{k} = \frac{0.44 - 1.75}{-0.072} = 18 \text{ weeks}$$

From the data shown in Table I, it appears that the predicted value would be in close agreement with the value actually obtained if an analysis had been done at this time.

With laboratory tests of this type, therefore, it should be possible to predict the changes resulting from storage with sufficient accuracy for surveillance purposes. It remains, however, to interpret the results of these tests in terms of safe life and functioning. In the former case, it appears that correlation with autoignition temperature would be a desirable criterion. In the latter case, it appears that correlation with firing, and, perhaps, closed bomb tests would be the criteria used to base predictions. At any rate, these analytical methods appear to have predicting power in regard to the property measured. There is now a great need to determine the relationship between the properties measured by chemical analyses and the actual performance in order to extend the predictive power of the laboratory tests.

PROCEDURE

1. Specimen

The specimen shall consist of approximately 5 gm of ground propellant weighed to within 0.2 mg.

2. Apparatus

2.1 Extractor (Roweg, Soxhlet or equivalent).

2.2 Extraction Thimble (Whatman, 22mm X 80mm).

2.3 Extraction flask

2.4 Condenser (Allihn type or equivalent).

2.5 Hot plate (preferably steam or hot water heater).

2.6 Steam distillation apparatus (fig. 3).

2.7 Spectrophotometer capable of producing monochromatic light in the specified visible region. This procedure assumes that the optical path of the spectrophotometric cell is 1 cm.

3. Material

3.1 Spectrophotometric standard:

(a) 2-Nitrodiphenylamine, MIL-N-3399A. If necessary, the material can be further purified by recrystallizing a saturated solution in absolute ethanol at room temperature by cooling in an ice-water bath. Separate the crystals by filtering through a Buchner funnel and pressing dry with a rubber sheet under vacuum. Dry the orange, needle-shape crystals in a vacuum dessiccator.

3.2 Methylene Chloride - MIL-D-6998 or equivalent.

3.3 Ethanol MIL-E-463, grade 1, or any equivalent grade of 95 percent ethyl alcohol.

3.4 Sodium Hydroxide (NaOH) Solution-Dissolve sufficient NaOH (American Chemical Society grade) in distilled water to give 15 g of solute per 100 ml of solution.

4. Procedure

4.1 Determination of absorptivity:

(a) Add approximately 0.125 g of the standard 2-Nitrodiphenylamine to a 250 ml volumetric flask; dissolve in and dilute to volume with ethanol. Transfer 2,3,4 and 5 ml aliquots from the ethanol solution to separate 100 ml volumetric flasks. Dilute each to volume with ethanol.

(b) Determine the absorptivity of each of the four solutions at 430 millimicrons (mu) by substituting in the following equation:

$$a = A/c$$

Where a = absorptivity

A = absorbance

c = concentration, mg/100 ml

Use the average obtained for the four dilutions as the absorptivity of the 2-Nitrodiphenylamine at this wavelength. If the standard deviation of the average exceeds 0.01, the determination should be repeated until this precision is attained.

NOTE

In all calculations use an absorbance value that has been corrected for difference between the cells used for the sample solution and for the reference (blank) solution. Determine this correction by filling all the cells with ethanol and measuring the absorbance at 430 mu after balancing the spectrophotometer against the reference cell. Ideally, the sample cell, or cells, should also be in balance, but, if not, it is most convenient to arrange the cells so that the sample cell, or cells have an absorbance that is positive with respect to the reference cell. With this arrangement, the difference between sample and reference cells is subtracted from the absorbance of the sample solution.

5. Analysis of specimen:

(a) Transfer the accurately weighed specimen to the extraction thimble and add 200 ml of methylene chloride to the extraction flask. Assemble the extraction apparatus on the hot plate and adjust the temperature so that the solvent drips from the condenser at a rate of two to three drops per second. Extract for 12 to 15 hours, preferably overnight. When the extraction is completed, evaporate the solvent from the extraction flask using a stream of dry air.

(b) Transfer the dry extract to a 100 ml volumetric flask with the aid of ethanol and bring up to volume with ethanol. Pipet a 20 ml aliquot of the solution into the balloon flask of the steam distillation apparatus shown in figure (3) and add 200 ml of the sodium hydroxide solution. Pass steam through the balloon flask and collect 375 ± 25 ml of distillate at a rate of 7 to 9 ml/minute.

(c) Transfer the distillate quantitatively to a one liter volumetric flask with the aid of ethanol, cool to room temperature and dilute to volume with this solvent.

(d) Determine the absorbance of this solution at 430 mμ using ethanol in the reference cell. Calculate the 2-Nitrodiphenylamine concentration by substituting in the following equation:

$$2NDPA,\% = \frac{A}{aW} \, 100$$

Where:

A = absorbance

a = absorptivity

W = sample weight in final dilution, mgs.

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TABLE I

Effect of Storage on Viscosity

Storage Time, wks		Viscosity, relative					
	Storage Temp. °C	65.5			80		
	Lot No.	P-3	P-4	P-5	P-3	P-4	P-5
0		1.543	1.539	1.531	1.543	1.539	1.531
2		--	--	--	1.417	1.444	1.455
4		1.492	1.520	1.484	1.386	1.408	1.404
8		1.483	1.496	1.485	1.271	1.291	1.284
12		1.474	1.478	1.233	1.195	1.470	1.230
16		1.460	1.460	1.452	1.179	1.204	1.217
20		1.471	1.490	1.480	1.143	1.158	1.163
24		1.466	1.470	1.423	1.127	1.120	1.117
28		1.438	1.422	1.460	--	--	--
32		1.413	1.394	1.407	--	--	--
36		1.389	1.407	1.433	--	--	--
Slope		-0.004	-0.004	-0.001	-0.016	-0.016	-0.016
Y - Intercept		1.53	1.53	1.46	1.45	1.49	1.47

Equation $V_t = kt \neq V_o$

TABLE II

Effect of Storage on Stabilizer Content

Storage Time, wks		$2NO_2DPA$, %					
	Storage Temp°, C	65.5			80		
	Lot No.	P-3	P-4	P-5	P-3	P-4	P-5
0		1.62	1.72	1.75	1.62	1.72	1.75
2		-	-	-	1.45	1.62	1.63
4		1.56	1.67	1.69	1.40	1.44	1.48
8		1.49	1.62	1.65	1.27	1.35	1.15
12		1.38	1.49	0.76	1.04	1.49	1.04
16		1.36	1.40	1.56	0.52	0.50	0.60
20		1.34	1.37	1.43	0.15	0.14	0.11
24		1.25	1.31	1.41	0.10	0.13	0.15
28		1.14	1.25	1.40			
32		1.15	1.19	1.36			
36		1.24	1.22	1.27			
40		1.13	1.18	1.25			
44		1.06	1.10	1.13			
48		0.84	1.05	0.92			
52		0.95	0.92	0.95			
Slope		-0.014	-0.014	-0.013	-0.068	-0.072	-0.072
Y - intercept		1.62	1.68	1.73	1.67	1.82	1.77

Equation $C_t = kt \not+ C_o$

Fig. 1 EFFECT OF STORAGE ON VISCOSITY

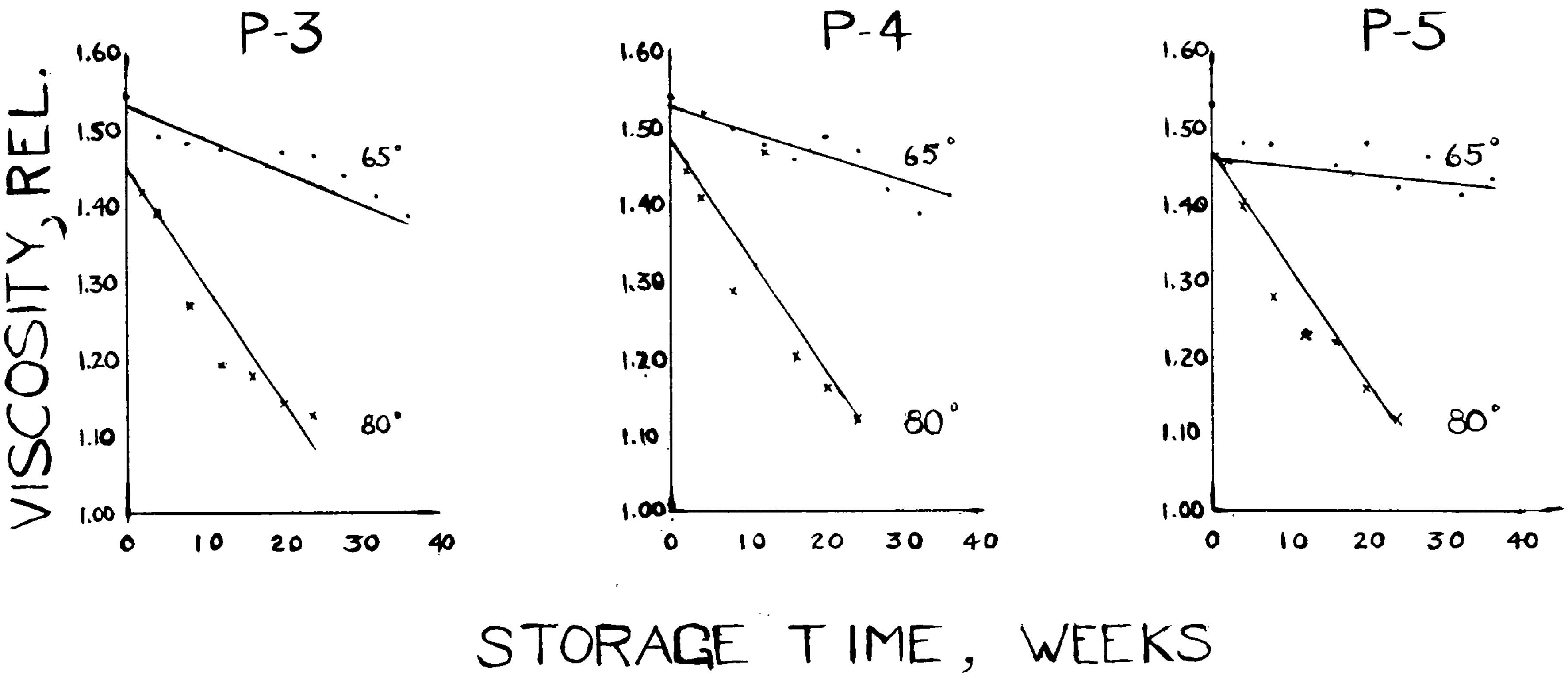


FIG. 2 EFFECT OF STORAGE ON STABILIZER

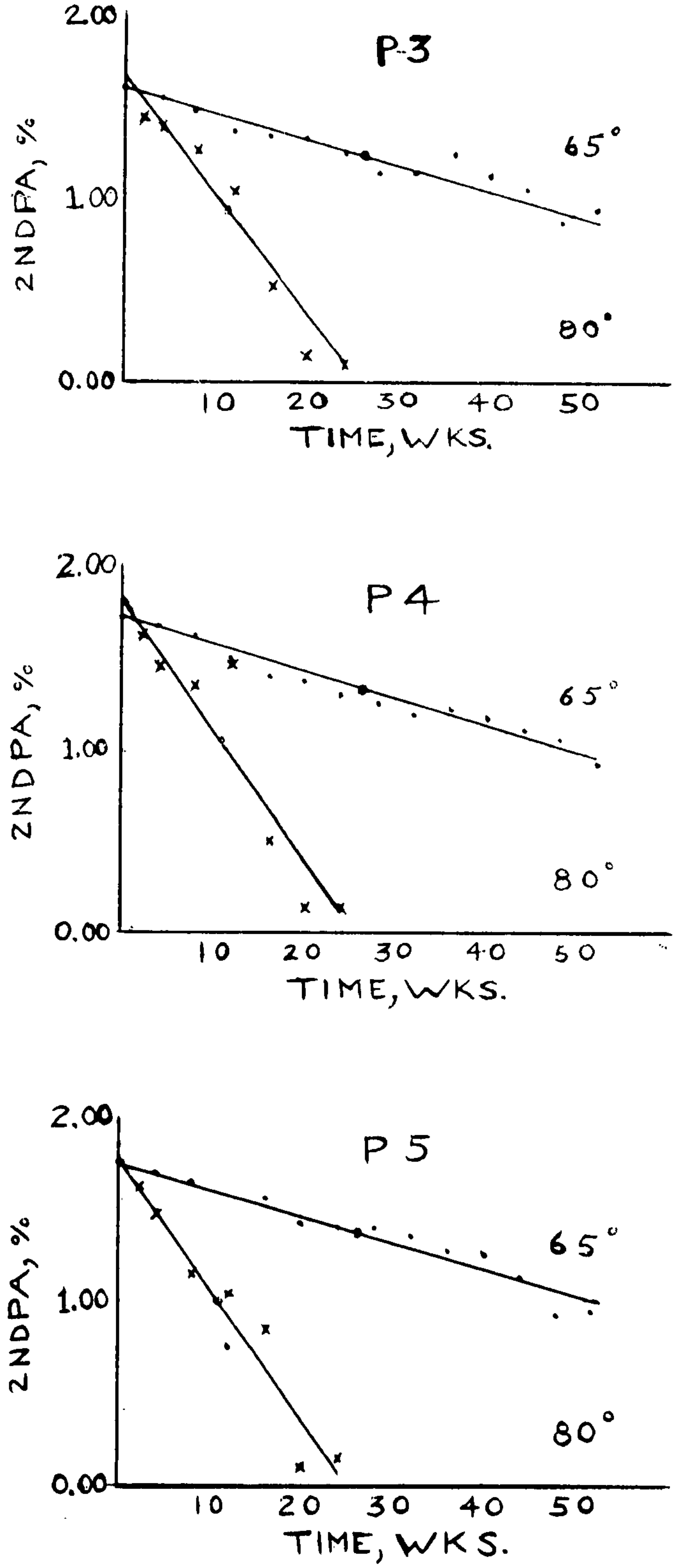
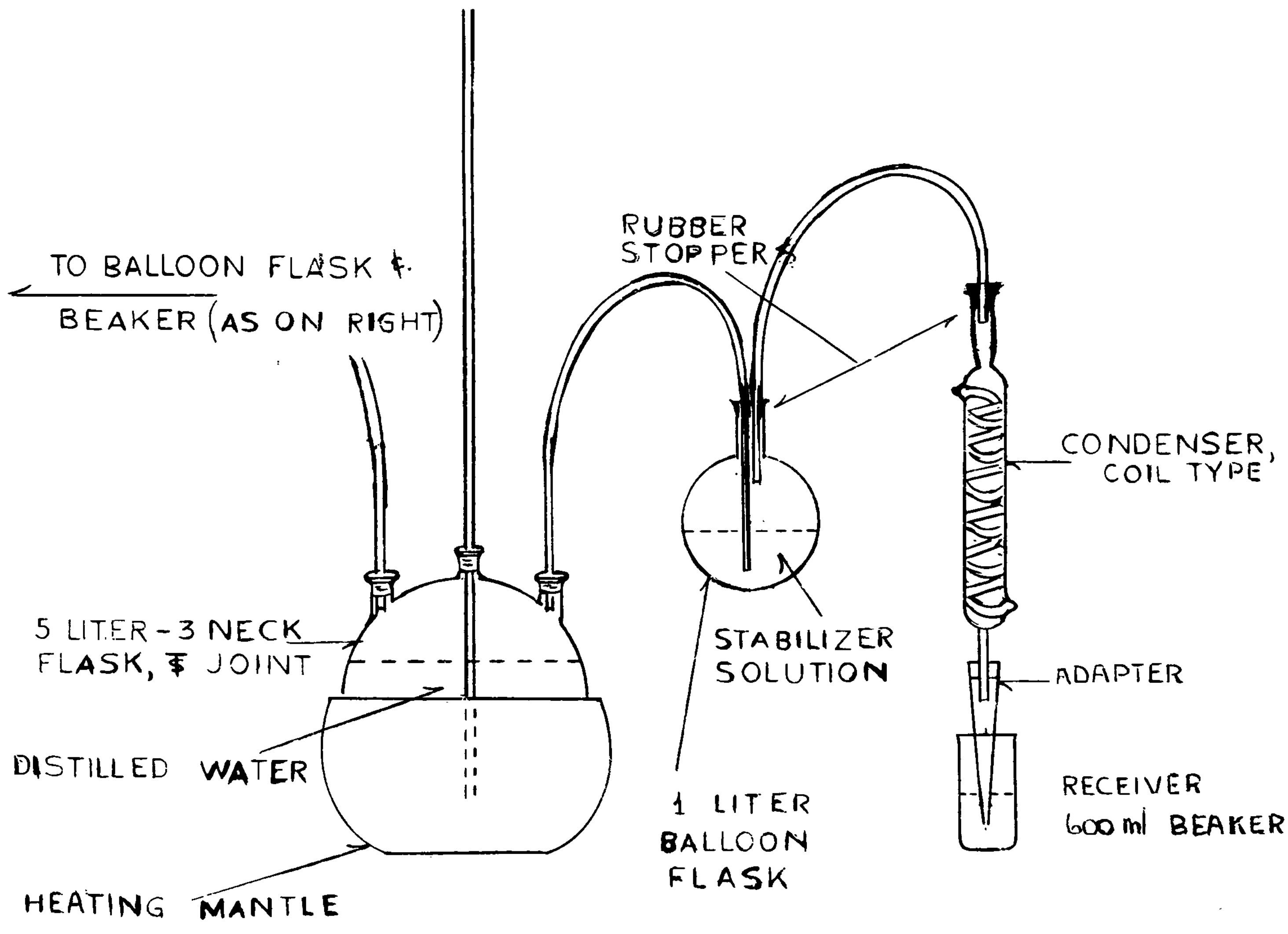


FIGURE 3

STEAM DISTILLATION APPARATUS



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Joint Army-Navy-Air Force Panel for
Analytical Chemistry of Solid Propellants

ABSTRACT

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